Sandia National Laboratories
Waste Isolation Pilot Plant (WIPP)
Test Plan, TP 00-02

Collection and Analysis of Downhole Cement And Steel Samples During Borehole Plugging and Abandonment

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1.0 Approval Page

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2.0 Table of Contents

3.0 Revision History

This is the first issue of this test plan (TP). Changes to TP 00-02, other than those defined as editorial changes per Nuclear Waste Management Program procedure NP 20-1, "Test Plans," shall be reviewed and approved by the same level of responsibility of persons that performed the original review and approval. All TP 00-02 revisions will follow the same distribution as the original document.

4.0 Definition Of Abbreviations And Acronyms

ASTM	American Society for Testing and Materials
CCA	Compliance Certification Application
DAS	Data acquisition system
EBSD	Electron backscatter diffraction
JCPDS-ICDD	Joint Committee on Powder Diffraction Standards - International Center for Diffraction Data
M&TE	Measuring and Test Equipment
MOC	Management and Operations Contractor
NIST	National Institute of Standards and Technology
NAS	National Academy of Sciences
NBS	National Bureau of Standards
NP	Nuclear Waste Management Program Procedure
NWMP	Nuclear Waste Management Program
P&A	Plugging and abandonment
RWP	Radiological Work Permit
SEM	Scanning Electron Microscope
SNL	Sandia National Laboratories
SP	Activity/Project Specific Procedure
TP	Test Plan
USI	Ultrasonic well-logging instrument
WID	Westinghouse Waste Isolation Division
WIPP	Waste Isolation Pilot Plant
XRD	X-ray Diffraction

5.0 Purpose and Scope

Upon reviewing the technical and scientific data gathered by Sandia National Laboratories (SNL) for the Waste Isolation Pilot Plant (WIPP) site characterization study, a National Academy of Sciences (NAS) review panel concluded that the only credible scenarios resulting in significant releases from the site involve human intrusion. Oil and gas deposits are common in the rock units underlying the repository formation (the Salado Formation), and currently active oil and gas wells are present within hundreds of meters of the site boundary (WIPP Compliance Certification Application (CCA), Appendix DEL). Inadvertent and intermittent drilling for resources is assumed to be the most severe human intrusion scenario. Estimates based on current and previous Delaware Basin drilling rates suggest that, over the next 10,000 years, 46.8 boreholes/km^2 will be drilled to a depth great enough to intersect the WIPP repository (CCA, Appendix DEL, Section 7.4).

Oil wells drilled within the Delaware Basin are plugged using several different plug configurations. These plug configurations are discussed in detail in the CCA, Appendix MASS, section 16. The three most prevalent types of plug configurations are:

1) A two-plug configuration, with one plug at the top of the Salado, between the water-bearing Rustler units and the repository and the other at the base of the evaporite sequence, below the Castile. The plugs are on average 40 m long, and are located at casing step-downs.

2) A three-plug configuration, that includes the two-plug configuration described in (1), plus a third plug in the Salado, either above or below the repository level. The plugs beneath the repository level are of particular importance, as they would serve to isolate the repository from brine pockets in the Castile.

3) A single-plug, extending through much or all of the evaporite section.

In the CCA, failure of borehole plugs is a function of two processes—corrosion of the steel casing and degradation of the cement comprising the plugs. Plugs at the top of the Salado were assumed to fail in less than 200 years due to casing corrosion. Those beneath the level of the repository were assumed to have failure lifetimes of greater than 5000 years and were assumed to maintain integrity over the regulatory lifetime of the repository (10,000 years). On the basis of this decision, units beneath the Castile were screened out as sources of brine for repository influx.

Samples of annular casing cement and concrete borehole plugs will test these assumptions and allow refinement of the conceptual model for concrete plug degradation presented in the CCA. In that document, concrete plugs above the Salado are assumed to fail rapidly due to casing failure, resulting in lack of axial confinement and axial infiltration of reactive brines leading to fracturing and spalling. Deeper plugs fall into two categories. Concrete plugs extending through the evaporite sequence are assumed to behave as confined systems, with alteration products building up within the plug and causing a decrease in porosity and permeability. Shorter plugs were treated as open systems, with porosity and permeability remaining constant until a “critical volume”—assumed from work by Berner (1990) to be 100 pore volumes—of water passes through the plug. At that point, concrete components will be degraded significantly, and unconstrained microfracturing and physical failure occur.

Concrete samples collected during plugging and abandonment (P&A) could examine several of these assumptions. Samples from cement plugs in the Rustler may indicate that these do not fail as quickly as estimated in the CCA, if expansive concrete alteration products do not form, or if reactions with magnesium, carbonate, and sulfate-saturated waters result in mineral precipitation (of brucite, calcite, or gypsum, respectively) on the concrete surface, armoring it against further reaction. Side-hole core samples can also be collected, providing a cross section of the casing and the annular casing cement. These might show, for example, that expansive casing corrosion products fracture the casing cement, and that flow through the fractured annular cement is a more probable mode of plug failure.

Similar effects may control plug failure at greater depths. In the CCA, steel well casing was assumed to corrode to form goethite ($\alpha FeOOH$). This reaction generates H_2, which, at repository depths and below, was assumed to build up in solution and inhibit further corrosion. Formation of a different Fe-oxyhydroxide, such as magnetite (Fe_3O_4) or "green rust" (a mixture of ferrous and ferric iron oxides/oxychlorides, observed by Telender and Westerman, (1997) under simulated WIPP repository conditions), would result different rates of H_2 generation, and different estimates of casing corrosion. More extensive corrosion might also occur if H_2 is consumed by microbial action, a possibility not considered in the CCA. Alternatively, precipitation of iron oxyhydroxides, carbonates, sulfides, or sulfates may passivate the metal surface and reduce or eliminate further corrosion. Examination of corrosion products and texture might also provide insights into the mechanism of corrosion—e.g., cathodic or microbially-mediated corrosion. Precipitated minerals may inhibit cement degradation reactions, as well (Krumhansl et al., 1993). Carbonate-rich Castile brines may be especially effective at promoting calcite precipitation and sealing cement porosity. Alternatively, cement plug degradation may be faster than predicted in the CCA, as the "critical volume" estimate of 100 pore volumes is based upon work with dilute solutions (Berner, 1990), rather than the complex, high ionic strength Castile and Salado brines.

During P&A of water-sampling boreholes at the WIPP site, there will be several opportunities for sampling casing and other steel parts (tubing, packers, etc), as well as casing cement and cement plugs. These materials have been in contact with supra-, and in some cases, sub-Salado brines for periods of up to 20 years. Examination and characterization of these materials should yield information on metal corrosion reactions and reaction rates, and cement/brine reactions under downhole conditions. Identification of the corrosion and degradation mechanisms, and the resultant mineralogical and textural changes in the materials, will provide information for better evaluating the predicted lifetimes of borehole plugs in intrusion boreholes. Data from downhole cement samples will support laboratory work being done to develop a more rigorous chemical model for cement degradation under WIPP-relevant conditions. Metal corrosion information derived from sampling during P&A will be useful for both the borehole plug longevity studies and as an analogue for metal corrosion within the repository.

The majority of boreholes scheduled for P&A offer the only opportunity to sample casing and cement from above the Salado. Such sampling may be useful in examining the general assumptions of plug failure in the CCA (casing and cement degradation mechanisms), and the assumption that casing corrosion products filling the borehole will have hydraulic properties similar to "unconsolidated sand." However, it is borehole plug failure at depths below the repository that potentially has the greatest impact on repository intrusion scenarios, as it is the failure of those plugs that ultimately allows brine entry and repository flooding. Of the holes scheduled for P&A, only one—WIPP-12—has a cement plug exposed to sub-Salado brines. This well contains a ~60 meter thick cement plug at the top of the Castile. The top of this plug has been exposed to Salado brines, and the bottom to Castile brines, for 20 years. The bottom-hole plugs in other holes are at the Salado/Rustler boundary. Thus, WIPP-12 is the only opportunity to examine cement degradation by Castile brine *in situ*. Ideally, a core of the entire cement plug would be collected; if this was precluded for safety reasons, then only the top of the plug might be cored. Although the hole is not cased at the level of the cement plug, a bridge plug was emplaced below the cement plug, and if this bridge plug is retrieved (necessary if hydrologic testing on the brine reservoir is to be performed), then steel components of the plug, exposed to H_2S-rich Castile brine for 20 years, could be collected for corrosion studies.

Collecting cement plug and steel samples from WIPP-12 and hydrologically testing the underlying Castile brine reservoir will be a complex procedure due to the safety issues involved. As such , it is beyond the scope of this test plan. If sampling WIPP-12 is mandated, a separate test plan will be issued.

6.0 Experimental Process Description

6.1 Planning Overall Strategy and Process

The basic procedure for these experiments will be to collect casing and concrete samples at the well site; to prepare them for chemical, mineralogical, or textural analysis by cutting, polishing, crushing, or dissolution; and to characterize them using a variety of analytical equipment.

6.1.1 Sampling

Well hole plugging and abandonment is done in several steps, as described below:

1) Pull tubing, packers, bridge plugs, and any other components from the hole.
2) Scrape and recirculate to remove scale from the inside of the hole. The scraping is done with a tube-shaped metal blade. On wells plugged to date, there was considerable scale build-up within the casing. In hole P-14, the 5 ½” diameter casing was plugged too less than 1” at a depth of 31’. In hole WIPP-28, there were two intervals, the first at 100’, where a 3 ½” tool was unable to pass through the 5 ½” casing. Ultrasonic logs for these wells indicate considerable thinning due to corrosion, with all the joints having at least local corrosion penetration of 30–50%. It seems probable that much of the observed scale is composed of iron oxyhydroxides and other casing corrosion products.
3) Tag the bottom of the hole to verify that the hole has been cleaned to the bottom.
4) Lower an ultrasonic probe (USI) to the bottom of the hole and log upward through the water-bearing zone. At the top of the water-bearing zone, deploy a bridge plug, fill the well with fresh water, and log to the top of the hole (this is necessary because the ultrasonic probe needs an aqueous medium to work properly).
5) Pull the bridge plug at the top of the water-bearing zone, replace the bridge plugs between the perforated zones to prevent co-mingling, and wait for evaluation of the ultrasonic core log to be completed.
6) If the log indicates that the cement-casing bond is good, and all regulatory agencies agree, then pull all bridge plugs, leave the casing in the hole, and cement back to the surface with

class C (sulfate-resistant) cement. Or, if the USI logs indicate a bad bond between the cement and the casing, pull the casing and cement the empty hole back to the surface.

There are several opportunities to obtain samples of downhole materials during this procedure.

1) Samples will be collected prior to any P&A activity, by sending a slick-line sampling device down the borehole. This device can collect grab-samples from the bottom of the hole before the hole is recirculated and cleaned. Such bottom samples will contain sloughed off scale materials, and may contain small pieces of concrete knocked into the hole during perforating, or other operations. Collection of this material will only allow us to evaluate corrosion products under downhole conditions, and to examine the texture and composition of the borehole filling material. It is assumed in the CCA that the borehole will be filled with corrosion scale from the upper parts of the borehole, and that this scale will have a porosity and permeability similar to that of unconsolidated sand. This assumption has never been tested.

2) Samples of steel and iron from the borehole will be collected from tubing, packers, and bridge plugs, as they are pulled from the hole. The bridge plugs and packers consist largely of stainless steel and non-metallic components, but there are some carbon steel components, and this is the material that will be sampled. Collecting pieces of corroded metal will allow us to examine the corrosion rind in cross section, from the metal surface to the interface of the rind with the brine.

3) Samples of the scale from the inside of the casing will be obtained during scraping and recirculation by collecting and filtering the brine as it is pumped out of the hole. Large pieces of scale, if present, will be obtained from the screens over the mud pit.

4) Once the hole has been scraped and cleaned, two opportunities for sampling exist. A small number of the holes scheduled for P&A have a cement plug at the bottom, and a core sample of the plug can be collected using a core drill. This is an important opportunity to collect cement, which has been exposed to formation fluids under down-hole conditions for extended periods of time. In addition, for those few holes, which have an inner diameter of greater than 7”, a side-bore sampler can be inserted into the hole and core samples taken through the casing and casing cement. This offers a unique opportunity to not only examine the casing and casing cement in cross section, but also the casing/cement and cement/wallrock boundaries, areas where preferential brine flow is likely to occur. Both of these sampling procedures (side-bore sampler and bottom-plug core) require placing a rotary rig over the hole, and are very expensive. However, only a few of the proposed holes meet the criteria for either of these sampling methods.

5) If the USI logging tool indicates that the casing cement bond is inadequate, then some or all of the casing string will be pulled from the hole prior to emplacing the cement plug. If this occurs, then samples of the casing and any casing cement adhering to it will be collected directly from the casing string using a diamond-coring bit.

The WIPP-site Management and Operations Contractor (MOC) is in charge of P&A operations (Richardson and Crawley, 1999), and P&A sampling will be a coordinated effort between SNL and the MOC. They plan to plug 4-6 wells per year, and have divided the candidate wells into high and low priority groups for P&A and placed them in a preliminary order for closure. This information is given in Table 1, along with an approximate schedule (assuming 5 wells per year),

Table 1. Plugging and Abandonment Schedule for WIPP-site water wells, and downhole sampling opportunities.

Well ID	Planned P&A Date	Possible downhole sampling opportunities
Group I (high priority) wells		
WIPP-28	Partially or fully completed, FY99	N/A
D268		N/A
P-14		N/A
H4a	FY00?	
H7a		
H10C		Collect a bailer sample from the top of the packer at 1459' after retrieval of the tubing.
H19b1		
WIPP-26		Collect a bailer sample from the top of the packer at 269'.
WIPP-12	FY01?	Collect a bailer sample from bridge plug at 1002.8'. Collect side-hole coring samples in Rustler and Salado levels, and cement core samples from the bottom hole plug at 2784'. Hydrologic tests on Castile brine pocket?
WIPP-27	FY01?	Collect a bailer sample from the top of the bridge plug at 399', and another from the top of the packer at 267' during P&A.
WIPP-29		Collect a bailer sample from the top of the bridge plug at 75'.
P-18		Collect a bailer sample from the top of the bridge plug at 997', and another from the top of the packer at 900' during P&A. Collect cement core samples from bottom-hole cement plug at 1125'.
H-8c		
Group II (lower priority) wells		
H2a	FY01?	
H2c	FY02?	Collect a bailer sample from the top of the bridge plug at 663.5'.
H3b3		
H5a		
H6a		
H7b1		
H7c	>FY02?	Collect a bailer sample from the top of the bridge plug at 305'.
H9a		
H11b1		
H11b2		
H11b3		
H14		
H18		Collect cement core samples from bottom-hole plug at 766'.
WIPP-13		Collect a bailer sample from the top of the bridge plug at 945'. Collect side-hole coring samples in Rustler and Salado levels.
WIPP-18		Collect cement core samples from bottom-hole plug at 1050'.
WIPP-22		Collect cement core samples from bottom-hole plug at 941'.
DOE-1		Collect a bailer sample from the top of the bridge plug at 891'.

and downhole sampling opportunities available for each hole. Scale samples and samples from retrieved downhole components will be collected for all holes, additional sampling will be done where appropriate. If a hole has been cased to the bottom, bottom-hole sediment samples will be collected using a slickline bailer. If it has an inner diameter of greater than 7", it will be considered for side-hole coring and if the bore hole has a cement bottom plug, taking core samples of that plug will be considered.

Initial sampling will consist of using a slickline sampling truck to collect bottom-hole samples from as several (~5) holes with a bailer. The slickline bailer can be run through tubing emplaced in the hole, but cannot be run down the annulus between the tubing and the casing. Samples can be collected from the bottom on the hole, or from the top of packers present in the hole. Holes chosen for this should be cased to the proposed sampling depth; otherwise, rock fragments sloughing off the walls of the borehole will dominate the bottom-hole sediment. As no damage will be done to the holes, this type of sampling need not be restricted to those scheduled for P&A. However, a time window exists for sampling the P&A holes, so they are given first priority. When tubing, packers, and other down-hole components are retrieved from a hole, a Sandia designated representative will be present onsite to describe the components pulled from the hole. Visual descriptions of the material will be entered into field notebooks, and samples of scale and metal will be taken from the components using a hack saw or a drill with a diamond-coring bit. Samples will be collected and stored according to NWMP procedures NP/SP 13-1, "Sample Control/Chain of Custody."

Sampling during scraping and recirculation will be coordinated with the MOC. Samples of the recirculated water will be filtered, and the filtrate will be collected and placed in a clean, properly labeled container. The recirculating water will also be screened, and larger pieces of scale will be collected from the screen. All activities will be documented in field notebooks according to NWMP procedure NP 20-2, "Scientific Notebooks." Pieces of corroded or cement encrusted casing and steel tubing may be collected with a hack-saw or diamond coring drill bit, to be sectioned and polished, and examined in cross section.

For either cement bottom-plug coring or side-bore coring, Sandia must arrange for a rotary drill rig to be moved onto the hole and a Sandia designated representative will be present to collect and log all samples retrieved. Sample descriptions (color, grain-size, texture, magnetic properties, etc.) will be recorded in field notebooks, and samples will be immediately placed in a pre-weighed plastic bottle or plastic bag labeled with a unique sample identification number. The headspace of the container will be purged with nitrogen, and it will be sealed, labeled, and placed in a second, labeled nitrogen-filled container. Upon delivery to the laboratory, the sample will be weighed, and transferred to a nitrogen-filled glove box to remain in storage for analysis. Following collection, samples will be prepared and examined by optical microscope, Scanning Electron Microscope (SEM), X-ray Diffraction (XRD), and bulk analysis.

6.1.2 Laboratory Analysis

Metal and scale samples and cement samples recovered from the boreholes during P&A will be characterized with respect to composition, mineralogy, and texture using one or more analytical techniques.

6.1.2.1 *Chemical analysis*:

Some scale and cement samples will be crushed, digested in acid, and analyzed using wet chemical techniques to determine the chemical composition of the material. Analysis will be accomplished with a Perkin Elmer Optima 3300 Dual View ICP, and with a Cary 300 UV-Visible spectrophotometer following Activity/Project Specific (SP) procedures (to be developed) for each instrument. Portions of cement and corrosion samples may be crushed and analyzed for carbonate content using a UIC, Inc. carbon analyzer.

6.1.2.2 *Textural and mineralogical characterization*:

Samples will be examined with a variety of tools to determine textural and mineralogical characteristics:

- Sieving. Scale samples retrieved from the bottom of the hole will be sieved and sized to test the “unconsolidated sand” hypothesis in the CCA. Sieving will be done in accordance with ASTM Standard Test Method C136-96a, “Standard Test Method for Sieve Analysis of Fine and Coarse Aggregates,” or an equivalent procedure.
- X-ray diffraction analysis. Scale and cement samples will be crushed and analyzed by X-ray diffraction using a Bruker AXS D8 Advance X-ray Spectrometer. If samples show evidence of being unstable in the atmosphere (exhibiting a change in color or texture upon exposure to atmosphere), they will be crushed and analyzed in an inert atmosphere (Ar or N_2). Very fine-grained samples may be sealed in a capillary tube and analyzed using a capillary stage.
- Characterization by SEM and optical microscopy. Samples will be sectioned and polished, and examined with an Olympus petrographic microscope with fluorescent capabilities, or with a JEOL JSM5900LV Scanning Electron Microscope (SEM). Sample preparation for SEM and optical microscope examination may include vacuum impregnation with low viscosity and/or fluorescent epoxy to examine porosity. The SEM is equipped with an energy-dispersive X-ray analysis system for qualitative compositional analysis of mineral grains and for multi-element mapping, and with an electron backscatter diffraction (EBSD) detector, for measuring the crystal structure of individual mineral grains. The structural information can be cross-referenced to the Joint Committee on Powder Diffraction Standards–International Centre for Diffraction Data (JCPDS-ICDD) database for phase identification purposes. Variations in sample mineralogy, texture and porosity will be described and photographed (digitally), and quantified if possible using Image Pro, an image processing and analysis software package.

6.2 Data Control

A calibration program will be implemented for the work described in this test plan in accordance with NP 12-1, “Control of Measuring and Test Equipment.” This M&TE calibration program will meet the requirements in NWMP procedure NP 12-1 for: (1) receiving and testing M&TE; (2) technical operating procedures for M&TE; (3) the traceability of our standards to nationally recognized standards such as those from the National Institute of Standards and Technology; (4) maintaining calibration records. In addition, NP 13-1and SP 13-1 identify requirements and appropriate forms for documenting and tracking sample possession.

6.2.1 Data Quality Control

Data collection procedures are specific to individual instruments. For details of the data acquisition for a particular instrument, see the specific SP or Users Manual for that instrument. A list of the relevant SPs is provided in Section 6.3. Any data acquired by a data acquisition system (DAS) will be attached directly to the Scientific Notebook or compiled in separate loose leaf binders with identifying labels to allow cross reference to the appropriate Scientific Notebook. If the instrument allows data to be recorded electronically, copies of the data disks will be submitted to the NWMP Records Center according to NWMP procedure NP 17-1 “Records.” For instruments that do not have direct data printout, the instrument readings will be recorded directly into the scientific notebook. Current versions of the DAS software will be included in the SNL WIPP Baseline Software List, as appropriate.

Quality control of the Scientific Notebooks will be established by procedures described in NWMP procedure NP 20-2 “Scientific Notebooks.” Methods for justification, evaluation, approval, and documentation of deviation from test standards and establishment of special prepared test procedures will be documented in the Scientific Notebooks. General procedures, goals and quality assurance controls for TP 00-02 are described below. Procedures including use of replicates, spikes, split samples, control charts, blanks and reagent controls will be determined during the development of experimental techniques as described in Section 6.1 above.

6.2.2 Data Acquisition Plan

The approach for collecting data varies for each instrument being used. Equipment data printouts will be attached directly to the scientific notebook or submitted to the NWMP Record Center. For instruments which do not have direct data printout (balances, pH meters), the instrument reading will be directly recorded in the scientific notebook. Data acquisition procedures for each instrument will follow the guidelines listed in the specific SP or TOP for that instrument. If no SP exists, or the analysis procedure listed in the SP is modified, the new procedure will be recorded in the scientific notebook.

The numerical data will be transferred from data printouts and scientific notebooks to Microsoft Excel (Office 97 version) spreadsheets. Data transfer and reduction shall be performed in such a way to ensure that data transfer is accurate, that no information is lost in the transfer, and that the input is completely recoverable. Data transfer and reduction shall be controlled to permit independent reproducibility by another qualified individual. A copy of each spreadsheet will be taped into the scientific notebook, and a second person will compare the data recorded in the notebook and that on the spreadsheet to verify that no transcription errors have occurred during technical and/or QA review of the notebook. This verification will be documented in the notebook when it is “signed off” by the reviewer.

6.2.3 Data Identification and Use

All calculations performed as part of the activities of TP 00-02 will be documented in a scientific notebook. The notebook will be technically reviewed periodically by a second person, who will note concurrence by co-signing the examined material. If a discrepancy is found, that discrepancy and its resolution will be documented in the notebook. In addition, there will be periodic quality assurance reviews of the notebook to ensure that the requirements of NWMP procedure NP 20-2, “Scientific Notebooks” are addressed.

Data generated under this TP will be used to evaluate the casing corrosion and cement alteration models used in the CCA, and to develop a more robust borehole plug degradation model for conditions at the WIPP site. In addition, downhole corrosion of steel borehole casing may be a suitable analog for steel corrosion within the WIPP repository, and yield information on near-field corrosion pathways and redox conditions.

6.3 Equipment

A variety of measuring and analytical equipment will be used for the work described in this test plan. This equipment includes that listed below, as well as equipment not yet purchased. A complete equipment list, including serial numbers, will be maintained in the scientific notebook. Much of the instrumentation to be used for this project is newly purchased, and operating procedures have not yet been developed or written. Scientific notebooks will be used to record all laboratory work activities.

Measuring and analytical equipment to be used for this project include:

6.3.1 Weighing Equipment.

Several balances are present in the facility and may be used for this project. These include a Mettler AT-261 five-decimal place electronic balance, an ANC three-decimal place balance, and top loading balances and scales with maximum ranges of 2 to 30 kilograms. Balance calibration checks will be performed routinely using the following NBS-traceable weight sets, which, in turn, are calibrated by the SNL Calibration Laboratory every 3 years:

- Troemner Calibration weight set, ASTM Class 1, Serial number 22803, 1 mg – 100 g, calibration expires 12/16/02.
- *Troemner Calibration weight*, NBS-Class 1, Serial number 42795, 100 g, calibration expires 11/19/02.
- *Troemner Calibration weight*, NBS-Class 1, Serial number 42797, 100 g, calibration expires 11/19/02.
- *Troemner Calibration weight*, NBS-Class 1, Serial number 42799, 100 g, calibration expires 11/19/01.
- *Troemner Calibration weight*, NBS-Class 1, Serial number 42800, 100 g, calibration expires 11/19/01.
- *Troemner Calibration weight*, ASTM-Class 1, Serial number 47824, 200 g, calibration expires 11/19/02.
- *Troemner Calibration weight*, ASTM-Class 1, Serial number 55335, 1000 g, calibration expires 11/19/02.
- *Troemner Calibration weight*, ASTM-Class 2, Serial number I-12, 10 kg, calibration expires 12/17/02.

Balance accuracy and precision will be checked daily or prior to use (whichever is less frequent), using the calibration weight sets listed above. Calibration checks will be recorded in the scientific notebook.

6.3.2 Liquid Measuring Equipment

Standard Laboratory Class A glassware (pipettes, volumetric flasks, etc.) will be used at all times. In addition, several adjustable Eppendorf pipettes, listed below, are available for use in the laboratory. The calibration of pipettes will be checked routinely against a calibrated balance, and will be recorded in the scientific notebook.

Pipette		Accuracy	Precision
0.5 – 10 µl	at 0.5 µ*l*	± 5 %	≤ 2.8 %
	at 5 µ*l*	±1.5 %	≤ 0.8 %
	at 10 µ*l*	± 1 %	≤ 0.4 %
2.0 – 20 µl	at 2.0 µ*l*	± 5 %	≤ 1.5 %
	at 10 µ*l*	± 1.2 %	≤ 0.6 %
	at 20 µ*l*	± 1.0 %	≤ 0.3 %
10 – 100 µl	at 10 µ*l*	±2.5 %	≤ 0.7 %
	at 50 µ*l*	±0.8 %	≤ 0.3 %
	at 100 µ*l*	± 0.8 %	≤ 0.15 %
50 – 200 µl	at 50 µ*l*	± 1 %	≤ 0.3 %
	at 100 µ*l*	± 0.9 %	≤ 0.3 %
	at 200 µ*l*	± 0.6 %	≤ 0.2 %
100 – 1000 µl	at 100 µ*l*	± 1.6 %	≤ 0.3 %
	at 1000 µ*l*	± 0.6 %	≤ 0.2 %
500 – 5000 µl	at 500 µ*l*	± 2.4 %	≤ 0.6 %
	at 5000 µ*l*	± 0.6 %	≤ 0.15 %
Eppendorf repeater plus: Accuracy and precision vary with tip used and amount dispensed. Tip sizes vary from 100 µl to 50 ml in 8 steps. Accuracy and precision for the largest and smallest are:			
	100µ*l* tip, 2µ*l* dispensed	± 1.6%	≤ 3.0 %
	100µ*l* tip, 20 µ*l* dispensed	± 1.0%	≤ 2.0 %
	50 m*l* tip, 1 m*l* dispensed	± 0.3%	≤ 0.5 %
	50 m*l* tip, 10 m*l* dispensed	± 0.3%	≤ 0.2 %

6.3.3 Other Analytical Equipment

- *pH Meters and Autotitrators* – solution pH may be measured using pH meters and/or autotitrators. A *Mettler Model MA235 pH/Ion Analyzer* and a *Mettler Model DL25 Autotitrator* will be used for this purpose. The range for all pH meters is 0.00 to 14.00. Electrodes will be calibrated before each use or daily (whichever is less frequent) with pH 4, 7, and 10 buffers manufactured by Fisher Scientific with unique lot numbers and expiration dates; traceable to the National Institute of Standards and Technology (NIST). The accuracy of the buffers is ±0.01 pH units; buffer values will be adjusted for laboratory temperatures as per buffer instruction sheets if necessary. Calibration checks will be recorded in the scientific notebook. Measuring pH in concentrated brines is difficult, and a procedure will be developed to calibrate pH meters.

- *Equipment for Chemical Analysis* – Three instruments may be used to chemically characterize crushed or digested samples of cement or casing scale. The first is a Perkin Elmer Optima 3300 DV Inductively-Coupled Plasma Optical Emission Spectrometer (ICP); the second is a Cary 300 UV-Visible Spectrophotometer; and the third, is a UIC, Inc. Carbon Analyzer, consisting of an acidification module, a furnace module, and a CO_2 coulometer. These instruments will be user-calibrated each time they are used and documented in the scientific notebook.
- *Equipment for Petrographic, and Textural Characterization* – Several instruments will be used for physical characterization of the samples collected during P&A of WIPP boreholes. Particle size distributions will be determined using ASTM-certified sieves. The mineralogy and texture may be characterized using either an Olympus BX60 Polarizing Microscope or a JEOL JSM 5900LV scanning electron microscope (SEM). Calibration standards will be used to verify instrument magnification when these instruments are used. Bulk sample mineralogy will be determined using a Bruker AXS D-8 Advance X-Ray Diffractometer (XRD). A mineral standard will be run periodically to verify diffraction line positions. Calibration results will be documented in the scientific notebook.

NMWP Activity/Project Specific Procedures (SPs) will be written for these instruments as necessary. Until that time, detailed procedure descriptions will be documented in laboratory notebooks.

6.4 Location and Personnel

All experimental work related to TP 00-02 will be performed at the SNL Carlsbad Operations laboratory facility located in Carlsbad, NM (laboratory work) or at the WIPP facility (sample collection). Sandia Personnel who include Charles Bryan and Mary-Alena Martell, and Sandia Contractors Ron Parsons and Wes DeYonge will carry out the work.

7.0 Nuclear Waste Management Program Procedures (NPs), and NWMP Activity/Project Specific Procedures (SPs)

The following technical work documents cover the work described in this Test Plan.

SOP-C001 – "Standard Operating Procedure for Activities in the SNL/Carlsbad Laboratory Facility."

SP 13-1 – "Chain of Custody."

NP 6-1 – "Document Review Process."

NP 13-1 – "Sample Control."

NP 12-1 – “Control Of Measuring And Test Equipment.”

NP 20-2 – “Scientific Notebooks.”

NP 2-1 – “Qualification and Training.”

NP 17-1 – “Records.”

In addition, SPs will be written for use of the ICP, SEM, XRD, Carbon analyzer, UV-Vis spectrophotometer, and assorted balances and scales used in the laboratory. Sample preparation procedures, which may vary from sample to sample as work scope evolves, will be detailed in Scientific Notebooks, in accordance with NWMP procedure NP 20-2. The latest versions of these documents are available on the internet at:

http://www.nwmp.sandia.gov/onlinedocuments/

8.0 Records, Reports, and Audits

We will consider all records providing evidence of quality, including but not necessarily limited to personnel qualification and training forms, lists of M&TE and software, technical procedures, laboratory notebooks, calibration records, and reports, to be QA records. We will maintain these records in accordance with NP 17-1, “Records.” We will, to the maximum extent possible, use the format of the enclosed WIPP Records Package to organize QA records. We may have records that do not fit into the categories of the WIPP Records Package, but we will follow this format as closely as possible. All of these records will be accurate, complete, identifiable, and legible. We will inspect them to ensure they satisfy these requirements prior to submittal to the NWMP Records Center. We will submit two copies of all QA records to the NWMP Records Center.

We will prepare documents for review and approval in accordance with NWMP procedure NP 6-1, “Document Review Process.” NP 6-1 requires that the author(s) and reviewer(s): (1) use the DRC Form NP 6-1-1, (see Appendix A in NP 6-1) in some, but not all, cases; (2) resolve all of the comments; (3) return this form with all signatures to the SNL/WIPP NWMP Records Center.

We will also review documents prepared by others in accordance with NP 6-1.

SNL/WIPP and DOE/CAO representatives will have the right to review the work described in this Test Plan.

9.0 Training

All personnel involved in the experiments described in TP 00-02 will be trained and qualified for their assigned work. This requirement will be implemented through NWMP procedure NP 2-1, “Qualification and Training.” Evidence of training to assigned NPs, SPs, TOPs, TP 00-02, ES&H procedures, and any other required training will be documented through Form NP 2-1-1 *Qualification and Training.* Annual Refresher QA training will ensure on-site personnel are trained to the NWMP QA Program.

10.0 Health and Safety

All of the health and safety requirements relevant to the work described in TP 00-02 and the procedures that will be used to satisfy these requirements are described in ES&H standard operating procedures. Sample collection at the WIPP site will be carried out following the site-specific ES&H procedures. SP-C001 (see section 7.1 above) describes the non-radiological hazards associated with these experiments and describes the procedures to deal with those hazards, including all the training requirements for personnel involved in conducting the experiments. In addition, a Radiological Work Permit (RWP) will be written for procedures involving use of the X-Ray Diffractometer. Additional SPs and RWPs may be mandated by SNL ES&H requirements and their issuance will not require revision of this Test Plan.

11.0 Permitting/Licensing

There are no special licenses or permit requirements for the work described in TP 00-02, as the well-head activities fall under the purview of the WID plugging and abandonment program.

12.0 References

Berner, U., 1990, “A Thermodynamic Description of the Evolution of Pore Water Chemistry and Uranium Speciation During the Degradation of Cement,” *Technical Report 90-12*, Paul Scherrer, Villigen, Switzerland.

Krumhansl, J.L., 1993, “The Accelerated Testing of Cement in Brines” *Proc. Am. Ceramic Soc., Cement-Based Materials*, April 19-22, 1993, Cincinnati, OH, p. 153-154.

Richardson, R.G., and M.E. Crawley, 1999, *Waste Isolation Pilot Plant Borehole Plugging and Abandonment Program Plan*, 36 pp. plus figures.